

DV Qualifies  
in database  
-HR

checked by IT  
2/27/17

# CETIFICATION

SDG No: FA40754 Laboratory: Accutest, Massachusetts  
Site: BMSMC, Humacao, PR Matrix: Soil

**SUMMARY:** Soil/Aqueous samples (Table 1) were collected on the BMSMC facility, Humacao, PR. Samples were collected January 25 2017 and were analyzed in Accutest Laboratory of Orlando, Florida that reported the data under SDG No.: FA40754. Results were validated using the following quality control criteria of the methods employed (MADEP VPH and MAPED EPH, Massachusetts Department of Environmental Protection, 2004) and the latest validation guidelines (July, 2015) of the EPA Hazardous Waste Support Section. The analyses performed are shown in Table 1. Individual data review worksheets are enclosed for each target analyte group. The data sample organic data samples summary form shows for analytes results that were qualified.

In summary the results are valid and can be used for decision taking purposes.

Table 1. Samples analyzed and analysis performed

SAMPLE ID	SAMPLE DESCRIPTION	MATRIX	ANALYSIS PERFORMED
FA40754-1	FTFSS-1	Soil	Volatiles TPHC Ranges Extractable TPHC Ranges
FA40754-2	FTFSS-2	Soil	Volatiles TPHC Ranges Extractable TPHC Ranges
FA40754-3	FTFSS-3	Soil	Volatiles TPHC Ranges Extractable TPHC Ranges
FA40754-4	B5SS-1	Soil	Volatiles TPHC Ranges Extractable TPHC Ranges
FA40754-5	B5SS-2	Soil	Volatiles TPHC Ranges Extractable TPHC Ranges
FA40754-6	EB-012517	AQ - Equipment Blank	Extractable TPHC Ranges
FA40754-7	FB-012517	AQ - Field Blank Soil	Volatiles TPHC Ranges Extractable TPHC Ranges

Reviewer Name: Rafael Infante  
Chemist License 1888

Signature:

*Rafael Infante*

Date:

February 16, 2017



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## Report of Analysis

Page 1 of 1

Client Sample ID:	FTFSS-1	Date Sampled:	01/25/17
Lab Sample ID:	FA40754-1	Date Received:	01/27/17
Matrix:	SO - Soil	Percent Solids:	93.3
Method:	MADEP VPH REV 1.1		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	UV077759.D	1	01/31/17	AJC	n/a	n/a	GUV4134
Run #2							

Run #	Initial Weight	Final Volume	Methanol Aliquot
Run #1	4.76 g	5.1 ml	100 ul
Run #2			

## MADEP VPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	6100	2100	ug/kg	
	C9- C12 Aliphatics (Unadj.)	ND	6100	2100	ug/kg	
	C9- C10 Aromatics (Unadj.)	ND	6100	2100	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
460-00-4	BFB	102%		70-130%
460-00-4	BFB	97%		70-130%



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

Page 1 of 1

Client Sample ID:	FTFSS-1	Date Sampled:	01/25/17
Lab Sample ID:	FA40754-1	Date Received:	01/27/17
Matrix:	SO - Soil	Percent Solids:	93.3
Method:	MADEP EPH REV 1.1 SW846 3546		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	NN017358.D	1	02/08/17	MG	02/01/17	OP63645	GNN877
Run #2							

Run #	Initial Weight	Final Volume
Run #1	19.7 g	2.0 ml
Run #2		

## MAEPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	ND	11000	8200	ug/kg	
	C9-C18 Aliphatics	ND	11000	5400	ug/kg	
	C19-C36 Aliphatics	ND	11000	5400	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
3386-33-2	1-Chlorooctadecane	61%		40-140%
580-13-2	2-Bromonaphthalene	78%		40-140%
84-15-1	o-Terphenyl	65%		40-140%
321-60-8	2-Fluorobiphenyl	87%		40-140%



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 N = Indicates presumptive evidence of a compound

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## Report of Analysis

Page 1 of 1

Client Sample ID:	FTFSS-2	Date Sampled:	01/25/17
Lab Sample ID:	FA40754-2	Date Received:	01/27/17
Matrix:	SO - Soil	Percent Solids:	88.9
Method:	MADEP VPH REV 1.1		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	UV077760.D	1	01/31/17	AJC	n/a	n/a	GUV4134
Run #2							

Run #	Initial Weight	Final Volume	Methanol Aliquot
Run #1	4.87 g	5.1 ml	100 ul
Run #2			

## MADEP VPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	6500	2300	ug/kg	
	C9- C12 Aliphatics (Unadj.)	ND	6500	2300	ug/kg	
	C9- C10 Aromatics (Unadj.)	ND	6500	2300	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
460-00-4	BFB	98%		70-130%
460-00-4	BFB	93%		70-130%



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

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 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

Page 1 of 1

Client Sample ID:	FTFSS-2	Date Sampled:	01/25/17
Lab Sample ID:	FA40754-2	Date Received:	01/27/17
Matrix:	SO - Soil	Percent Solids:	88.9
Method:	MADEP EPH REV 1.1 SW846 3546		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	NN017405.D	1	02/10/17	MG	02/01/17	OP63645	GNN879
Run #2							

Run #	Initial Weight	Final Volume
Run #1	20.1 g	2.0 ml
Run #2		

## MAEPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	10200	11000	8400	ug/kg	J
	C9-C18 Aliphatics	ND	11000	5600	ug/kg	
	C19-C36 Aliphatics	19100	11000	5600	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
3386-33-2	1-Chlorooctadecane	50%		40-140%
580-13-2	2-Bromonaphthalene	67%		40-140%
84-15-1	o-Terphenyl	52%		40-140%
321-60-8	2-Fluorobiphenyl	71%		40-140%



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 RL = Reporting Limit  
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J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

Page 1 of 1

Client Sample ID:	FTFSS-3	Date Sampled:	01/25/17
Lab Sample ID:	FA40754-3	Date Received:	01/27/17
Matrix:	SO - Soil	Percent Solids:	94.9
Method:	MADEP VPH REV 1.1		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	UV077768.D	1	02/01/17	AJC	n/a	n/a	GUV4135
Run #2							

Run #	Initial Weight	Final Volume	Methanol Aliquot
Run #1	3.07 g	5.1 ml	100 ul
Run #2			

## MADEP VPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	9000	3200	ug/kg	
	C9- C12 Aliphatics (Unadj.)	ND	9000	3200	ug/kg	
	C9- C10 Aromatics (Unadj.)	ND	9000	3200	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
460-00-4	BFB	94%		70-130%
460-00-4	BFB	89%		70-130%



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

Page 1 of 1

Client Sample ID:	FTFSS-3	Date Sampled:	01/25/17
Lab Sample ID:	FA40754-3	Date Received:	01/27/17
Matrix:	SO - Soil	Percent Solids:	94.9
Method:	MADEP EPH REV 1.1 SW846 3546		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	NN017361.D	1	02/08/17	MG	02/01/17	OP63645	GNN877
Run #2							

Run #	Initial Weight	Final Volume
Run #1	20.2 g	2.0 ml
Run #2		

## MAEPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	13600	10000	7800	ug/kg	
	C9-C18 Aliphatics	ND	10000	5200	ug/kg	
	C19-C36 Aliphatics	ND	10000	5200	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
3386-33-2	1-Chlorooctadecane	67%		40-140%
580-13-2	2-Bromonaphthalene	80%		40-140%
84-15-1	o-Terphenyl	68%		40-140%
321-60-8	2-Fluorobiphenyl	86%		40-140%



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 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

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 B = Indicates analyte found in associated method blank  
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## Report of Analysis

Page 1 of 1

Client Sample ID: B5SS-1  
 Lab Sample ID: FA40754-4  
 Matrix: SO - Soil  
 Method: MADEP VPH REV 1.1  
 Project: BMSMC, Humacao, PR

Date Sampled: 01/25/17  
 Date Received: 01/27/17  
 Percent Solids: 93.5

	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	UV077769.D	1	02/01/17	AJC	n/a	n/a	GUV4135
Run #2							

	Initial Weight	Final Volume	Methanol Aliquot
Run #1	5.03 g	5.1 ml	100 ul
Run #2			

## MADEP VPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	5800	2000	ug/kg	
	C9- C12 Aliphatics (Unadj.)	ND	5800	2000	ug/kg	
	C9- C10 Aromatics (Unadj.)	ND	5800	2000	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
460-00-4	BFB	99%		70-130%
460-00-4	BFB	94%		70-130%



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound



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## Report of Analysis

Page 1 of 1

Client Sample ID:	B5SS-1	Date Sampled:	01/25/17
Lab Sample ID:	FA40754-4	Date Received:	01/27/17
Matrix:	SO - Soil	Percent Solids:	93.5
Method:	MADEP EPH REV 1.1 SW846 3546		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	NN017362.D	1	02/08/17	MG	02/01/17	OP63645	GNN877
Run #2							

Run #	Initial Weight	Final Volume
Run #1	20.1 g	2.0 ml
Run #2		

## MAEPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	ND	11000	8000	ug/kg	
	C9-C18 Aliphatics	ND	11000	5300	ug/kg	
	C19-C36 Aliphatics	ND	11000	5300	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
3386-33-2	1-Chlorooctadecane	68%		40-140%
580-13-2	2-Bromonaphthalene	74%		40-140%
84-15-1	o-Terphenyl	67%		40-140%
321-60-8	2-Fluorobiphenyl	80%		40-140%



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 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

Page 1 of 1

Client Sample ID:	B5SS-2	Date Sampled:	01/25/17
Lab Sample ID:	FA40754-5	Date Received:	01/27/17
Matrix:	SO - Soil	Percent Solids:	94.6
Method:	MADEP VPH REV 1.1		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	UV077770.D	1	02/01/17	AJC	n/a	n/a	GUV4135
Run #2							

Run #	Initial Weight	Final Volume	Methanol Aliquot
Run #1	4.06 g	5.1 ml	100 ul
Run #2			

## MADEP VPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	6900	2400	ug/kg	
	C9- C12 Aliphatics (Unadj.)	ND	6900	2400	ug/kg	
	C9- C10 Aromatics (Unadj.)	ND	6900	2400	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
460-00-4	BFB	96%		70-130%
460-00-4	BFB	91%		70-130%



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

Page 1 of 1

Client Sample ID:	B5SS-2	Date Sampled:	01/25/17
Lab Sample ID:	FA40754-5	Date Received:	01/27/17
Matrix:	SO - Soil	Percent Solids:	94.6
Method:	MADEP EPH REV 1.1 SW846 3546		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	NN017363.D	1	02/08/17	MG	02/01/17	OP63645	GNN877
Run #2							

Run #	Initial Weight	Final Volume
Run #1	20.7 g	2.0 ml
Run #2		

## MAEPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	ND	10000	7700	ug/kg	
	C9-C18 Aliphatics	ND	10000	5100	ug/kg	
	C19-C36 Aliphatics	ND	10000	5100	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
3386-33-2	1-Chlorooctadecane	67%		40-140%
580-13-2	2-Bromonaphthalene	73%		40-140%
84-15-1	o-Terphenyl	58%		40-140%
321-60-8	2-Fluorobiphenyl	80%		40-140%



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

Page 1 of 1

Client Sample ID:	EB-012517	Date Sampled:	01/25/17
Lab Sample ID:	FA40754-6	Date Received:	01/27/17
Matrix:	AQ - Equipment Blank	Percent Solids:	n/a
Method:	MADEP EPH REV 1.1 SW846 3510C		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	NN017292.D	1	02/03/17	MG	01/31/17	OP63636	GNN873
Run #2							

Run #	Initial Volume	Final Volume
Run #1	800 ml	2.0 ml
Run #2		

## MAEPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	ND	250	190	ug/l	
	C9-C18 Aliphatics	ND	250	130	ug/l	
	C19-C36 Aliphatics	ND	250	130	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
3386-33-2	1-Chlorooctadecane	47%		40-140%
580-13-2	2-Bromonaphthalene	82%		40-140%
84-15-1	o-Terphenyl	74%		40-140%
321-60-8	2-Fluorobiphenyl	87%		40-140%



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

Page 1 of 1

Client Sample ID:	FB-012517	Date Sampled:	01/25/17
Lab Sample ID:	FA40754-7	Date Received:	01/27/17
Matrix:	AQ - Field Blank Soil	Percent Solids:	n/a
Method:	MADEP VPH REV 1.1		
Project:	BMSMC, Humacao, PR		

	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	UV077880.D	1	02/07/17	AJC	n/a	n/a	GUV4141
Run #2							

	Purge Volume
Run #1	5.0 ml
Run #2	

## MADEP VPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	100	35	ug/l	
	C9- C12 Aliphatics (Unadj.)	ND	100	35	ug/l	
	C9- C10 Aromatics (Unadj.)	ND	100	35	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
460-00-4	BFB	92% <sup>a</sup>		70-130%
460-00-4	BFB	86% <sup>a</sup>		70-130%

(a) Surrogate recoveries corrected for actual spike amount.



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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 20 of 815  
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## Report of Analysis

Page 1 of 1

Client Sample ID: FB-012517  
 Lab Sample ID: FA40754-7  
 Matrix: AQ - Field Blank Soil  
 Method: MADEP EPH REV 1.1 SW846 3510C  
 Project: BMSMC, Humacao, PR

Date Sampled: 01/25/17  
 Date Received: 01/27/17  
 Percent Solids: n/a

	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	NN017293.D	1	02/03/17	MG	01/31/17	OP63636	GNN873
Run #2							

	Initial Volume	Final Volume
Run #1	800 ml	2.0 ml
Run #2		

## MAEPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	ND	250	190	ug/l	
	C9-C18 Aliphatics	ND	250	130	ug/l	
	C19-C36 Aliphatics	ND	250	130	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
3386-33-2	1-Chlorooctadecane	45%		40-140%
580-13-2	2-Bromonaphthalene	77%		40-140%
84-15-1	o-Terphenyl	68%		40-140%
321-60-8	2-Fluorobiphenyl	83%		40-140%



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J = Indicates an estimated value  
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 N = Indicates presumptive evidence of a compound



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FA40754 PAGE 1 OF 1

<div style="font-size: 2em; font-weight: bold; margin: 0;">FL</div>		4435 Vineyard Road, Suite C-15, Orlando, Florida 32811 TEL. 407-425-6700 FAX. 407-425-6707 www.acutest.com		L0437 <b>77623506710</b> Analytical Order # _____		Mobile Order Customer # _____ Analytical Order # _____			
		Client / Reporting Information		Project Information		Requested Analysis (see TEST CODE sheet)		Matrix Codes	
Company Name <b>Anderson Mulholland &amp; Associates</b> Street Address <b>2700 Westchester Avenue, Suite 417</b> City State Zip <b>NY 10577</b> Purchase Order # <b>814-251-0400</b> Project Contact <b>Terry Taylor</b> Phone # <b>814-251-0400</b> Sample(s) Name(s)		Project Name <b>Brink-Myers Squibb Manufacturing Co., Surface Soil Sampling</b> Street <b>Humacao</b> Project # <b>PR</b> Client Purchase Order # <b>Terry Taylor</b> Address City State Zip Abandon:		Billing Information (if different from Report to) Company Name Street Address City State Zip		DW - Drinking Water GW - Ground Water WW - Wastewater SW - Surface Water SO - Soil SL - Sludge SED - Sediment OL - Oil LIQ - Other Liquid AIR - Air SOL - Other Solid WIP - Wipes FB - Fecal Bacteria EB - Equipment Bacteria RB - Rinse Blank TB - Trip Blank		LAB USE ONLY	
Field ID / Point of Collection <b>1 FTFSS-1</b> <b>2 FTFSS-2</b> <b>3 FTFSS-3</b> <b>4 B5SS-1</b> <b>5 B5SS-2</b> <b>6 EB-012517</b> <b>7 EB-012517</b>		Collection Date Time Sampled by Matrix # of bottles <b>1/25/17 1045 TT SO 3</b> <b>1/25/17 1105 TT SO 3</b> <b>1/25/17 1110 TT SO 3</b> <b>1/25/17 1135 TT SO 3</b> <b>1/25/17 1525 TT SO 3</b> <b>1/25/17 0918 TT EB 828</b> <b>1/25/17 1456 TT FB 828</b>							
Turnaround Time (Business days) <input checked="" type="checkbox"/> Std. 10 Business Days <input type="checkbox"/> Std. 10 Business Days (by contract only) <input type="checkbox"/> 10 Day RUSH <input type="checkbox"/> 8 Day RUSH <input type="checkbox"/> 5 Day EMERGENCY <input type="checkbox"/> 2 Day EMERGENCY <input checked="" type="checkbox"/> 1 Day EMERGENCY Emergency & Rush T/A rates available via website		Approved By (Approved PRT) / Date _____ _____ _____ _____ _____		Data Deliverable Information <input type="checkbox"/> Commercial "A" (Level 1) <input type="checkbox"/> Commercial "B" (Level 2) <input checked="" type="checkbox"/> FULLT1 (Level 3+4) <input type="checkbox"/> NJ Reduced <input type="checkbox"/> Commercial "C" <input type="checkbox"/> NYASP Category A <input type="checkbox"/> NYASP Category B <input type="checkbox"/> State Forms <input type="checkbox"/> EDO Forms <input type="checkbox"/> Other		Comments / Special Instructions <b>Only report Aliphatic and Aromatic</b> <b>Concentrations Do not report individual</b> <b>PAHs</b>			
Repackaged by: <b>1/26/17</b>		Date Time: <b>1/26/17 7:00</b>		Repackaged by: <b>FedEx</b>		Date Time: <b>1/26/17</b>		Repackaged by: <b>1/26/17</b>	
Repackaged by: <b>1/26/17</b>		Date Time: <b>1/26/17</b>		Repackaged by: <b>1/26/17</b>		Date Time: <b>1/26/17</b>		Repackaged by: <b>1/26/17</b>	

**FA40754: Chain of Custody**  
**Page 1 of 3**

## EXECUTIVE NARRATIVE

SDG No: **FA40754** Laboratory: **Accutest, Florida**  
Analysis: **MADEP VPH** Number of Samples: **6**  
Location: **BMSMC, Humacao, PR**  
**Humacao, PR**

**SUMMARY:** Six (6) samples were analyzed for Volatiles TPHC Ranges by method MADEP VPH. Samples were validated following the METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDROCARBONS (VPH) quality control criteria, Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

**Critical issues:** **None**  
**Major:** **None**  
**Minor:** **None**

**Critical findings:** **None**  
**Major findings:** **None**  
**Minor findings:** 1. The % difference in the continuing and ending calibration verification for the C5-C8 hydrocarbon range outside the method performance criteria. Results qualified as estimated (J or UJ) in affected samples.  
  
2. MS/MSD % recovery and RPD within laboratory control limits except for the cases described in the Data Review Worksheet. MS/MSD results apply to the unspiked sample. Unspiked sample was from another job. No qualification performed based on MS/MSD.

**COMMENTS:** Results are valid and can be used for decision making purposes.

**Reviewers Name:** **Rafael Infante**  
**Chemist License 1888**

**Signature:**

A handwritten signature in blue ink, reading "Rafael Infante", is written over a horizontal line.

**Date:** **February 16, 2017**



## SAMPLE ORGANIC DATA SAMPLE SUMMARY

Sample ID: FA40754-1  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2017  
Matrix: Soil

### METHOD: MADEP VPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç5 - C8 Aliphatics (Unadj.)	6100	ug/kg	1	-	U	Yes
Ç9 - C12 Aliphatics (Unadj.)	6100	ug/kg	1	-	U	Yes
Ç9 - C10 Aromatics (Unadj.)	6100	ug/kg	1	-	U	Yes

Sample ID: FA40754-2  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2017  
Matrix: Soil

### METHOD: MADEP VPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç5 - C8 Aliphatics (Unadj.)	6500	ug/kg	1	-	U	Yes
Ç9 - C12 Aliphatics (Unadj.)	6500	ug/kg	1	-	U	Yes
Ç9 - C10 Aromatics (Unadj.)	6500	ug/kg	1	-	U	Yes

Sample ID: FA40754-3  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2016  
Matrix: Soil

METHOD: MADEP VPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç5 - C8 Aliphatics (Unadj.)	9000	ug/kg	1	-	U	Yes
Ç9 - C12 Aliphatics (Unadj.)	9000	ug/kg	1	-	U	Yes
Ç9 - C10 Aromatics (Unadj.)	9000	ug/kg	1	-	U	Yes

Sample ID: FA40754-4  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2017  
Matrix: Soil

METHOD: MADEP VPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç5 - C8 Aliphatics (Unadj.)	5800	ug/kg	1	-	U	Yes
Ç9 - C12 Aliphatics (Unadj.)	5800	ug/kg	1	-	U	Yes
Ç9 - C10 Aromatics (Unadj.)	5800	ug/kg	1	-	U	Yes

Sample ID: FA40754-5  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2017  
Matrix: Soil

METHOD: MADEP VPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç5 - C8 Aliphatics (Unadj.)	6900	ug/kg	1	-	U	Yes
Ç9 - C12 Aliphatics (Unadj.)	6900	ug/kg	1	-	U	Yes
Ç9 - C10 Aromatics (Unadj.)	6900	ug/kg	1	-	U	Yes

Sample ID: FA40754-7  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2017  
Matrix: AQ - Field Blank Soil

METHOD: MADEP VPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç5 - C8 Aliphatics (Unadj.)	100	ug/L	1	-	UJ ✓	Yes
Ç9 - C12 Aliphatics (Unadj.)	100	ug/L	1	-	U	Yes
Ç9 - C10 Aromatics (Unadj.)	100	ug/L	1	-	U	Yes

# DATA REVIEW WORKSHEETS

Type of validation Full: ☒ Limited: ☐ Project Number: FA40754 Date: 01/25/2017 Shipping date: 01/26/2017 EPA Region: 2

## REVIEW OF VOLATILE PETROLEUM HYDROCARBON (VPHs) PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to the data validation guidance documents in the following order of precedence METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDROCARBONS (VPH), Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest Laboratories - Orlando data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: FA40754 Sample matrix: Soil  
No. of Samples: 7  
Field blank No.: FA40754-7  
Equipment blank No.: -  
Trip blank No.: -  
Field duplicate No.: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input type="checkbox"/> GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input type="checkbox"/> Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input checked="" type="checkbox"/> Matrix Spike/Matrix Spike Duplicate	

Overall Comments:  
\_Volatiles\_by\_GC\_by\_Method\_MADEP\_VPH,\_REV\_1.1.

### Definition of Qualifiers:

J- Estimated results  
U- Compound not detected  
R- Rejected data  
UJ- Estimated nondetect

Reviewer: Rafael Infante  
Date: February 15, 2017

## DATA REVIEW WORKSHEETS

All criteria were met   x    
Criteria were not met and/or see below           

### I. DATA COMPLETNESS

#### A. Data Package:

MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED


#### B. Other

Discrepancies:


## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED	DATE ANALYZED	ACTION
Samples analyzed within method recommended holding time. Sample preservation within the required criteria.				

### Criteria

#### Preservation:

Samples analyzed with ambient purge temperature: Samples must be acidified to a pH of 2.0 or less at the time of collection.

Samples analyzed with heated purge temperature: Samples must be treated to a pH of 11.0 or greater at the time of collection.

Methanol preservation of soil/sediment samples is mandatory. Methanol (purge-and-trap grade) must be added to the sample vial before or immediately after sample collection. In lieu of the in-field preservation of samples with methanol, soil samples may be obtained in specially-designed air tight sampling devices, provided that the samples are extruded and preserved in methanol within 48 hours of collection.

#### Holding times:

Aqueous samples using ambient or heated purge - analyze within 14 days.

Soil/sediment samples - analysis within 28 days.

Cooler temperature (Criteria:  $4 \pm 2$  °C):       4.2°C      

#### Actions: Qualify positive results/non-detects as follows:

If holding times are exceeded, estimate positive results (J) and nondetects (UJ).

If holding times are grossly exceeded, use professional judgment to qualify data. The data reviewer may choose to estimate positive results (J) and rejects nondetects (R).

If samples were not at the proper temperature ( $> 10^{\circ}\text{C}$ ) or improperly preserved, use professional judgment to qualify the results.

# DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

## CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:   01/13/17  

Dates of initial calibration verification:   01/13/17  

Instrument ID numbers:           HP5890          

Matrix/Level:           AQUEOUS/MEDIUM          

DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED
Initial and initial calibration verification meet method specific requirements				

### Criteria- ICAL

- Five point calibration curve.
- The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest. When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.
- A collective calibration factor must also be established for each hydrocarbon range of interest. Calculate the collective CFs for C5-C8 Aliphatic Hydrocarbons and C9-C12 Aliphatic Hydrocarbons using the FID chromatogram. Calculate the collective CF for the C9-C10 Aromatic Hydrocarbons using the PID chromatogram. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest.

### Criteria- CCAL

- At a minimum, the working calibration factor must be verified on each working day, after every 20 samples, and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity.
- If the percent difference (%D) for any analyte varies from the predicted response by more than  $\pm 25\%$ , a new five-point calibration must be performed for that analyte. Greater percent differences are permissible for n-nonane. If the %D for n-nonane is greater than 30, note the nonconformance in the case narrative. It should be noted that the %Ds are calculated when CFs are used for the initial calibration and

## DATA REVIEW WORKSHEETS

percent drifts are calculated when calibration curves using linear regression are used for the initial calibration.

### Actions:

If %RSD > 25% for target compounds or a correlation coefficient < 0.99, estimate positive results (J) and use professional judgment to qualify nondetects.

If % D > 25% (> 30 for nonane), estimate positive results (J) and nondetects (UJ).

### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: \_\_\_\_\_ 01/13/17 \_\_\_\_\_

Dates of continuing calibration verification: \_\_ 01/31/17; \_02/06/17\_\_

Dates of final calibration verification: \_01/31/17; \_02/07/17\_\_\_\_\_

Instrument ID numbers: \_\_\_\_\_ HP5890 \_\_\_\_\_

Matrix/Level: \_\_\_\_\_ AQUEOUS/MEDIUM \_\_\_\_\_

DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED
02/06/17	cc4115-4	C5 – C8 Aliphatics	-29.6	FA40754-7
02/07/17	Cc4115-4	C5 – C8 Aliphatics	-24.9	

**Note:** Continuing and final calibration verification meets method specific requirements except in the cases described in this document. The % difference for VPH in the C5-C8 aliphatic hydrocarbon retention time window in the continuing and ending calibration verification was outside the method performance criteria. Results are qualified as estimated in affected samples.

A separate worksheet should be filled for each initial curve



# DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

## V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data. A Laboratory Method Blank must be run after samples suspected of being highly contaminated to determine if sample carryover has occurred.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

### Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_METHOD_BLANKS_MEET_THE_METHOD_SPECIFIC_CRITERIA_____				
_____				
_____				
_____				
_____				

**Note:**

### Field/Trip/Equipment

A methanol trip blank or acidified reagent water trip blank **should** continually accompany each soil/sediment sample or water sample batch, respectively, during sampling, storage, and analysis.

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_NO_TRIP/EQUIPMENT_BLANKS_ASSOCIATED_WITH_THIS_DATA_PACKAGE._____				
_NO_TARGET_ANALYTES_DETECTED_IN_FIELD_BLANK_ANALYZED._____				
_____				
_____				
_____				

**Note:**

## DATA REVIEW WORKSHEETS

### V B. BLANK ANALYSIS RESULTS (Section 3)

#### Blank Actions

The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent MCP cleanup standard. Specific actions area as follows:

If the concentration is  $<$  sample quantitation limit (SQL) and  $<$  AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but  $<$  AL, report the compound as not detected (U) at the reported concentration.

If the concentration is  $>$  AL, report the concentration unqualified.

# DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

## SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND	ACTION
	2,3,4-Trifluorotoluene	

  SURROGATE STANDARD RECOVERIES WITHIN LABORATORY CONTROL  
  LIMITS. SURROGATE RECOVERIES WERE CORRECTED FOR ACTUAL SPIKE  
  AMOUNT.

QC Limits\* (Aqueous)

       LL   to   UL          70  to  130          to               to       

QC Limits\* (Solid)

       LL   to   UL          70  to  130          to               to       

It is recommended that surrogate standard recoveries be monitored and documented on a continuing basis. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 70% or more than 130%, check calculations to locate possible errors, check the fortifying standard solution for degradation, and check changes in instrument performance.

If the cause cannot be determined, reanalyze the sample unless one of the following exceptions applies:

- (1) Obvious interference is present on the chromatogram (e.g., unresolved complex mixture);
- (2) Percent moisture of associated soil/sediment sample is >25% and surrogate recovery is >10%; or
- (3) The surrogate exhibits high recovery and associated target analytes or hydrocarbon ranges are not detected in sample.

If a sample with a surrogate recovery outside of the acceptable range is not reanalyzed based on any of these aforementioned exceptions, this information must be noted on the data report form and discussed in the Executive Report. Analysis of the sample on dilution may diminish matrix-related surrogate recovery problems. This approach can be used as long as the reporting limits to evaluate applicable MCP standards can still be achieved with the dilution. If not, reanalysis without dilution must be performed.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples.

At the request of the data user, and in consideration of sample matrices and data quality objectives, matrix spikes and matrix duplicates may be analyzed with every batch of 20 samples or less per matrix.

- **Matrix duplicate** - Matrix duplicates are prepared by analyzing one sample in duplicate. The purpose of the matrix duplicates is to determine the homogeneity of the sample matrix as well as analytical precision. The RPD of detected results in the matrix duplicate samples must not exceed 50 when the results are greater than 5x the reporting limit.
- The desired spiking level is 50% of the highest calibration standard. However, the total concentration in the MS (including the MS and native concentration in the unspiked sample) should not exceed 75% of the highest calibration standard in order for a proper evaluation to be performed. The purpose of the matrix spike is to determine whether the sample matrix contributes bias to the analytical results. The corrected concentrations of each analyte within the matrix spiking solution must be within 70 - 130% of the true value. Lower recoveries of n-nonane are permissible (if included in the calibration of the C9-C12 aliphatic range), but must be noted in the narrative if <30%.

#### MS/MSD Recoveries and Precision Criteria

Sample ID: FA40755-4\_MS/MSD

Matrix/Level: Soil

List the %Rs, RPD of the compounds which do not meet the QC criteria.

The QC reported here applies to the following samples:

Method: **MADEP VPH REV 1.1**

**FA40754-3, FA40754-4, FA40754-5**

Compound	FA40755-4 ug/kg	Q	Spike ug/kg	MS ug/kg	MS %	Spike ug/kg	MSD ug/kg	MSD %	RPD	Limits Rec/RPD
C5- C8 Aliphatics (Unadj.)	ND		36100	21000	58*	36100	20800	58*	1	70-130/50
C9- C12 Aliphatics (Unadj.)	ND		30100	10000	33*	30100	9970	33*	0	70-130/50
C9- C10 Aromatics (Unadj.)	ND		18100	5490	30*	18100	5080	28*	8	70-130/50

\* Outside laboratory control limits.

**Note:** MS/MSD % recovery and RPD within laboratory control limits except for the cases described in this document. MS/MSD results apply to the unspiked sample. Unspiked sample was from another job. No qualification performed based on MS/MSD.

## DATA REVIEW WORKSHEETS

No action is taken on MS/MSD results alone to qualify the entire case. However, used informed professional judgment, the data reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, the qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that the laboratory is having a systematic problem in the analysis of one or more analytes, which affects the associated samples.

### 2. MS/MSD – Unspiked Compounds

List the concentrations of the unspiked compounds and determine the % RSDs of these compounds in the unspiked sample, matrix spike, and matrix spike duplicate.

COMPOUND	CONCENTRATION		MSD	%RPD	ACTION
	SAMPLE	MS			

Criteria: None specified, use  $\%RSD \leq 50$  as professional judgment.

Actions:

If the  $\%RSD > 50$ , qualify the results in the spiked sample as estimate (J).

If the  $\%RSD$  is not calculable (NC) due to nondetect value in the sample, MS, and/or MSD, use professional judgment to qualify sample data.

A separate worksheet should be used for each MS/MSD pair.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### VIII. LABORATORY CONTROL SAMPLE (LCS/LCSD) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT	ACTION
--------	----------	-----	----------	--------

  LCS\_RECOVERY\_WITHIN\_LABORATORY\_CONTROL\_LIMITS  

#### Criteria:

- \* Refer to QAPP for specific criteria.
- \* The spike recovery must be between 70% and 130%. Lower recoveries of n-nonane are permissible (if included in the calibration of the C9-C12 aliphatic range). If the recovery of n-nonane is <30%, note the nonconformance in the executive narrative.

#### Actions:

Actions on LCS recovery should be based on both the number of compounds that are outside the %R criteria and the magnitude of the exceedance of the criteria.

If the %R of the analyte is > UL, qualify all positive results (j) for the affected analyte in the associated samples and accept nondetects.

If the %R of the analyte is < LL, qualify all positive results (j) and reject (R) nondetects for the affected analyte in the associated samples.

If more than half the compounds in the LCS are not within the required recovery criteria, qualify all positive results as (J) and reject nondetects (R) for all target analyte(s) in the associated samples.

#### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix (1 per 20 samples per matrix)? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected. Discuss the actions below:

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### IX. FIELD/LABORATORY DUPLICATE PRECISION

Sample IDs:   FA40754-2/FA40754-2DUP  

Matrix:   Soil  

Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measures only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
Laboratory duplicate analyzed with this data package. RPD within laboratory and validation guidance document criteria ( $\pm 50\%$ ) for analytes detected above reporting limits.					

#### Criteria:

The project QAPP should be reviewed for project-specific information.  
RPD  $\pm 30\%$  for aqueous samples, RPD  $\pm 50\%$  for solid samples if results are  $\geq$  SQL.  
If both samples and duplicate are  $< 5$  SQL, the RPD criteria is doubled.

SQL = soil quantitation limit

#### Actions:

If both the sample and the duplicate results are nondetects (ND), the RPD is not calculable (NC). No action is needed.

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria.

If one sample result is not detected and the other is  $\geq 5$ x the SQL qualify (J/UJ).

**Note:** If SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is  $< 5$ x the SQL, use professional judgment to determine if qualification is appropriate.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### XI. COMPOUND IDENTIFICATION

The compound identification evaluation is to verify that the laboratory correctly identified target analytes as well as tentatively identified compounds (TICs).

1. Verify that the target analytes were within the retention time windows.
  - Retention time windows must be re-established for each Target VPH Analyte each time a new GC column is installed, and must be verified and/or adjusted on a daily basis.
  - Coelution of the m- and p- xylene isomers is permissible.
  - All surrogates must be adequately resolved from individual Target Analytes included in the VPH Component Standard.
  - For the purposes of this method, adequate resolution is assumed to be achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks.
  - The n-pentane (C5) and MtBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively.

**Note:** Target analytes were within the retention time window.

2. If target analytes and/or TICs were not correctly identified, request that the laboratory resubmit the corrected data.



## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### XII. QUANTITATION LIMITS AND SAMPLE RESULTS

The sample quantitation evaluation is to verify laboratory quantitation results.

1. In the space below, please show a minimum of one sample calculation:

FID

Computer printout

PID

Computer printout

2. If requested, verify that the results were above the laboratory method detection limit (MDLs).
3. If dilutions performed, were the SQLs elevated accordingly by the laboratory? List the affected samples and dilution factor in the table below.

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION

If dilution was not performed and the results were above the concentration range, estimate results (J) for the affected compounds. List the affected samples/compounds:

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## EXECUTIVE NARRATIVE

SDG No: **FA40754** Laboratory: **Accutest, Florida**  
Analysis: **MADEP EPH** Number of Samples: **7**  
Location: **BMSMC, Humacao, PR**  
**Humacao, PR**

**SUMMARY:** Seven (7) samples were analyzed for Extractables TPHC Ranges by method MADEP EPH. Samples were validated following the METHOD FOR THE DETERMINATION OF EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) quality control criteria, Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

**Critical issues:** **None**  
**Major:** **None**  
**Minor:** **None**

**Critical findings:** **None**  
**Major findings:** **None**  
**Minor findings:** 1. MS/MSD % recovery and RPD within laboratory control limits except for the cases described in the Data Review Worksheet. MS/MSD results apply to the unspiked sample. Unspiked sample was from another job. No qualification performed based on MS/MSD.

**COMMENTS:** Results are valid and can be used for decision making purposes.

**Reviewers Name:** **Rafael Infante**  
**Chemist License 1888**

**Signature:**

A handwritten signature in blue ink, reading "Rafael Infante", is written over a horizontal line.

**Date:** **February 16, 2017**

## SAMPLE ORGANIC DATA SAMPLE SUMMARY

Sample ID: FA40754-1  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2017  
Matrix: Soil

### METHOD: MADEP EPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	11000	ug/kg	1	-	U	Yes
Ç9 - C18 Aliphatics	11000	ug/kg	1	-	U	Yes
Ç19 - C36 Aliphatics	11000	ug/kg	1	-	U	Yes

Sample ID: FA40754-2  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2017  
Matrix: Soil

### METHOD: MADEP EPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	10200	ug/kg	1	J	J	Yes
Ç9 - C18 Aliphatics	6500	ug/kg	1	-	U	Yes
Ç19 - C36 Aliphatics	19100	ug/kg	1	-	-	Yes

Sample ID: FA40754-3  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2016  
Matrix: Soil

METHOD: MADEP EPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	13600	ug/kg	1	-	-	Yes
Ç9 - C18 Aliphatics	10000	ug/kg	1	-	U	Yes
Ç19 - C36 Aliphatics	10000	ug/kg	1	-	U	Yes

Sample ID: FA40754-4  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2017  
Matrix: Soil

METHOD: MADEP EPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	11000	ug/kg	1	-	U	Yes
Ç9 - C18 Aliphatics	11000	ug/kg	1	-	U	Yes
Ç19 - C36 Aliphatics	11000	ug/kg	1	-	U	Yes

Sample ID: FA40754-5  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2017  
Matrix: Soil

METHOD: MADEP EPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	10000	ug/kg	1	-	U	Yes
Ç9 - C18 Aliphatics	10000	ug/kg	1	-	U	Yes
Ç19 - C36 Aliphatics	10000	ug/kg	1	-	U	Yes

Sample ID: FA40754-6  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2017  
Matrix: AQ - Equipment Blank

METHOD: MADEP EPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	250	ug/L	1	-	U	Yes
Ç9 - C18 Aliphatics	250	ug/L	1	-	U	Yes
Ç19 - C36 Aliphatics	250	ug/L	1	-	U	Yes

Sample ID: FA40754-7  
Sample location: BMSMC, Humacao, PR  
Sampling date: 1/25/2017  
Matrix: AQ - Field Blank Soil

METHOD: MADEP EPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	250	ug/L	1	-	U	Yes
Ç9 - C18 Aliphatics	250	ug/L	1	-	U	Yes
Ç19 - C36 Aliphatics	250	ug/L	1	-	U	Yes

# DATA REVIEW WORKSHEETS

Type of validation Full: ☒ Limited: ☐ Project Number: FA40754  
 Date: 01/25/2017  
 Shipping date: 01/26/2017  
 EPA Region: 2

## REVIEW OF EXTRACTABLE PETROLEUM HYDROCARBON (EPHs) PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to the data validation guidance documents in the following order of precedence METHOD FOR THE DETERMINATION OF EXTRACTABLE PETROLEUM HYDROCARBONS (EPH), Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest Laboratories - Orlando data package received has been reviewed and the quality control and performance data summarized. The data review for SVOCs included:

Lab. Project/SDG No.: FA40754 Sample matrix: Soil  
 No. of Samples: 7  
 Field blank No.: FA40754-7  
 Equipment blank No.: FA40754-6  
 Trip blank No.: -  
 Field duplicate No.: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input type="checkbox"/> GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input type="checkbox"/> Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input checked="" type="checkbox"/> Matrix Spike/Matrix Spike Duplicate	

Overall Extractable\_Petroleum\_Hydrocarbons\_by\_GC\_by\_Method\_MADEP\_EPH\_REV\_1.1 Comments:

### Definition of Qualifiers:

J- Estimated results  
 U- Compound not detected  
 R- Rejected data  
 UJ- Estimated nondetect

Reviewer: Rafael Defaut

Date: February 16, 2017

All criteria were met x

## I. DATA COMPLETNESS

**A. Data Package:**

### MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

[illegible]

B. Other

**Discrepancies:**

[illegible]



## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED	DATE ANALYZED	ACTION
Samples extracted and analyzed within method recommended holding time				

### Criteria

#### Preservation:

Aqueous samples must be acidified to a pH of 2.0 or less at the time of collection.

Soil samples must be cooled at  $4 \pm 2$  °C immediately after collection.

#### Holding times:

Samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

Cooler temperature (Criteria:  $4 \pm 2$  °C):   4.2°C  

#### Actions: Qualify positive results/nondetects as follows:

If holding times are exceeded, estimate positive results (J) and nondetects (UJ).

If holding times are grossly exceeded, use professional judgment to qualify data. The data reviewer may choose to estimate positive results (J) and rejects nondetects (R).

If samples were not at the proper temperature ( $> 10^{\circ}\text{C}$ ) or improperly preserved, use professional judgment to qualify the results.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met and/or see below   X  

### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 10/29/16

Dates of initial calibration verification: 10/29/16

Instrument ID numbers: FID\_7

Matrix/Level: AQUEOUS/MEDIUM

DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED
Initial and continuing calibration meet method specific requirements				

#### Criteria- ICAL

- Five point calibration curve.
- The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest. When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.
- A collective calibration factor must also be established for each hydrocarbon range of interest. Calculate the collective CFs for C9-C18 Aliphatic Hydrocarbons, C19-C36 Aliphatic Hydrocarbons, and C11-C22 Aromatic Hydrocarbons using the FID chromatogram. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest.
  - The area for the surrogates must be subtracted from the area summation of the range in which they elute.
  - The areas associated with naphthalene and 2-methylnaphthalene in the aliphatic range standard must be subtracted from the uncorrected collective C9-C18 Aliphatic Hydrocarbon range area prior to calculating the CF.

## DATA REVIEW WORKSHEETS

### Criteria- CCAL

- At a minimum, the working calibration factor must be verified on each working day, after every 20 samples or every 24 hours (whichever is more frequent), and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity.
- If the percent difference (%D) for any analyte varies from the predicted response by more than  $\pm 25\%$ , a new five-point calibration must be performed for that analyte. Greater percent differences are permissible for n-nonane. If the %D for n-nonane is greater than 30, note the nonconformance in the case narrative. It should be noted that the %Ds are calculated when CFs are used for the initial calibration and percent drifts are calculated when calibration curves using linear regression are used for the initial calibration.

### Actions:

If %RSD > 25% for target compounds or a correlation coefficient < 0.99, estimate positive results (J) and use professional judgment to qualify nondetects.

If % D > 25% (> 30 for nonane), estimate positive results (J) and nondetects (UJ).

### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: \_\_\_\_\_ 10/29/16 \_\_\_\_\_

Dates of continuing calibration verification: \_\_\_\_\_ 02/02/17; 02/08/17; 02/10/17 \_\_\_\_\_

Dates of final calibration verification: \_\_\_\_\_ 02/02/17; 02/08/17; 02/10/17 \_\_\_\_\_

Instrument ID numbers: \_\_\_\_\_ FID-7 \_\_\_\_\_

Matrix/Level: \_\_\_\_\_ AQUEOUS/MEDIUM \_\_\_\_\_

DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED
Initial and continuing calibration meets method specific requirements.				

### Note:

A separate worksheet should be filled for each initial curve.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data. A Laboratory Method Blank must be run after samples suspected of being highly contaminated to determine if sample carryover has occurred.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
------------------	--------	------------------	----------	------------------------

  METHOD BLANKS MEET THE METHOD SPECIFIC CRITERIA.  

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

**Note:**

Field/Trip/Equipment

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
------------------	--------	------------------	----------	------------------------

  NO TRIP BLANK ANALYZED ASSOCIATED WITH THIS DATA PACKAGE.  

  NO TARGET ANALYTES DETECTED IN FIELD/EQUIPMENT BLANK  

  ASSOCIATED WITH THIS DATA PACKAGE.  

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### V      B.      BLANK ANALYSIS RESULTS (Section 3)

#### Blank Actions

The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent MCP cleanup standard. Specific actions area as follows:

If the concentration is  $<$  sample quantitation limit (SQL) and  $<$  AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but  $<$  AL, report the compound as not detected (U) at the reported concentration.

If the concentration is  $>$  AL, report the concentration unqualified.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND				ACTION
	S1	S2	S3	S4	
<u>_SURROGATE_STANDARDS_RECOVERIES_WITHIN_LABORATORY_CONTROL_</u>					
<u>_LIMITS_</u>					

#### Note:

S1 = o-Terphenyl 40-140%

S2 = 2-Fluorobiphenyl 40-140%

S3 = 1-Chlorooctadecane 40-140%

S4 = 2-Bromonaphthalene 40-140%

QC Limits (%)\* (Aqueous)

  LL  to  UL     40  to  140     40  to  140     40  to  140     40  to  140  

QC Limits\* (Solid)

  LL  to  UL     40  to  140     40  to  140     40  to  140     40  to  140  

It is recommended that surrogate standard recoveries be monitored and documented on a continuing basis. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 40% or more than 140%, check calculations to locate possible errors, check the fortifying standard solution for degradation, and check changes in instrument performance.

If the cause cannot be determined, reanalyze the sample unless one of the following exceptions applies:

- (1) Obvious interference is present on the chromatogram (e.g., unresolved complex mixture);
- (2) The surrogate exhibits high recovery and associated target analytes or hydrocarbon ranges are not detected in sample.

If a sample with a surrogate recovery outside of the acceptable range is not reanalyzed based on any of these aforementioned exceptions, this information must be noted on the data report form and discussed in the Executive Report. Analysis of the sample on dilution may diminish matrix-related surrogate recovery problems. This approach can be used as long as the reporting limits to evaluate applicable MCP standards can still be achieved with the dilution. If not, reanalysis without dilution must be performed.

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met and/or see below \_\_\_\_\_

### VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples.

At the request of the data user, and in consideration of sample matrices and data quality objectives, matrix spikes and matrix duplicates may be analyzed with every batch of 20 samples or less per matrix.

- **Matrix duplicate** - Matrix duplicates are prepared by analyzing one sample in duplicate. The purpose of the matrix duplicates is to determine the homogeneity of the sample matrix as well as analytical precision. The RPD of detected results in the matrix duplicate samples must not exceed 50 when the results are greater than 5x the reporting limit.
- The desired spiking level is 50% of the highest calibration standard. However, the total concentration in the MS (including the MS and native concentration in the unspiked sample) should not exceed 75% of the highest calibration standard in order for a proper evaluation to be performed. The purpose of the matrix spike is to determine whether the sample matrix contributes bias to the analytical results. The corrected concentrations of each analyte within the matrix spiking solution must be within 40 - 140% of the true value. Lower recoveries of n-nonane are permissible but must be noted in the narrative if <30%.

#### MS/MSD Recoveries and Precision Criteria

Sample ID: FA40755-4\_MS/MSD Matrix/Level: Soil

List the %Rs, RPD of the compounds which do not meet the QC criteria.

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
<u>_MSD</u>	<u>C9 - C18 Aliphatics</u>	<u>36</u>		<u>40-140/50</u>	<u>No_action</u>

**Note:** No action taken. MS/MSD results apply to unspiked sample. Unspiked sample was from another job.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below       

No action is taken on MS/MSD results alone to qualify the entire case. However, used informed professional judgment, the data reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, the qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that the laboratory is having a systematic problem in the analysis of one or more analytes, which affects the associated samples.

### 2. MS/MSD – Unspiked Compounds

List the concentrations of the unspiked compounds and determine the % RSDs of these compounds in the unspiked sample, matrix spike, and matrix spike duplicate.

COMPOUND	CONCENTRATION		MSD	%RPD	ACTION
	SAMPLE	MS			

Criteria: None specified, use %RSD  $\leq$  50 as professional judgment.

Actions:

If the % RSD > 50, qualify the results in the spiked sample as estimate (J).

If the % RSD is not calculable (NC) due to nondetect value in the sample, MS, and/or MSD, use professional judgment to qualify sample data.

A separate worksheet should be used for each MS/MSD pair.



## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### VIII. LABORATORY CONTROL SAMPLE (LCS/LCSD) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT	ACTION
--------	----------	-----	----------	--------

  LCS\_RECOVERY\_WITHIN\_LABORATORY\_CONTROL\_LIMITS.  

Note:

Criteria:

- \* Refer to QAPP for specific criteria.
- \* The spike recovery must be between 40% and 140%. Lower recoveries of n-nonane are permissible. If the recovery of n-nonane is <30%, note the nonconformance in the executive narrative. RPD between LCS/LCSD must be < 25%.

Actions:

Actions on LCS recovery should be based on both the number of compounds that are outside the %R and RPD criteria and the magnitude of the exceedance of the criteria.

If the %R of the analyte is > UL, qualify all positive results (j) for the affected analyte in the associated samples and accept nondetects.

If the %R of the analyte is < LL, qualify all positive results (j) and reject (R) nondetects for the affected analyte in the associated samples.

If more than half the compounds in the LCS are not within the required recovery criteria, qualify all positive results as (J) and reject nondetects (R) for all target analyte(s) in the associated samples.

#### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix (1 per 20 samples per matrix)? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected. Discuss the actions below:

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## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### IX. FIELD/LABORATORY DUPLICATE PRECISION

Sample IDs:   FA40651-1/FA40651-1DUP   Matrix:   Aqueous    
Sample IDs:   FA40754-1/FA40754-1DUP   Matrix:   Soil  

Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measures only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
Laboratory duplicates analyzed with this data package. RPD within laboratory and generally acceptable control limits					

#### Criteria:

The project QAPP should be reviewed for project-specific information.  
RPD  $\pm$  30% for aqueous samples, RPD  $\pm$  50 % for solid samples if results are  $\geq$  SQL.  
If both samples and duplicate are  $< 5$  SQL, the RPD criteria is doubled.

SQL = soil quantitation limit

#### Actions:

If both the sample and the duplicate results are nondetects (ND), the RPD is not calculable (NC). No action is needed.

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria.

If one sample result is not detected and the other is  $\geq 5$ x the SQL qualify (J/UJ).

**Note:** If SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is  $< 5$ x the SQL, use professional judgment to determine if qualification is appropriate.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### XI. COMPOUND IDENTIFICATION

The compound identification evaluation is to verify that the laboratory correctly identified target analytes as well as tentatively identified compounds (TICs).

1. Verify that the target analytes were within the retention time windows.
  - Retention time windows must be re-established for each Target EPH Analyte each time a new GC column is installed, and must be verified and/or adjusted on a daily basis.
  - The n-nonane (n-C9) peak must be adequately resolved from the solvent front of the chromatographic run.
  - All surrogates must be adequately resolved from the Aliphatic Hydrocarbon and Aromatic Hydrocarbon standards.
  - For the purposes of this method, adequate resolution is assumed to be achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks.
  - The n-pentane (C5) and MtBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively.

- 1a. Aliphatic hydrocarbons range:
  - Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for n-C9 and 0.01 minutes before the Rt for n-C19.
  - Determine the total area count for all peaks eluting 0.01 minutes before the Rt for n-C19 and 0.1 minutes after the Rt for n-C36.

Are the aliphatic hydrocarbons range properly determined? Yes? or No?

Comments:

- 1b. Aromatic hydrocarbons range:
  - Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for naphthalene and 0.1 minutes after the Rt for benzo(g,h,i)perylene.
  - Determine the peak area count for the sample surrogate (OTP) and fractionation surrogate(s). Subtract these values from the collective area count value.

Are the aliphatic hydrocarbons range properly determined? Yes? or No?

Comments:

DATA REVIEW WORKSHEETS

All criteria were met   X  

Criteria were not met and/or see below           

2. If target analytes and/or TICs were not correctly identified, request that the laboratory resubmit the corrected data.
3. Breakthrough determination - Each sample (field and QC sample) must be evaluated for potential breakthrough on a sample specific basis by evaluating the % recovery of the fractionation surrogate (2-bromonaphthalene) and on a batch basis by quantifying naphthalene and 2-methylnaphthalene in both the aliphatic and aromatic fractions of the LCS and LCSD. **If either the concentration of naphthalene or 2-methylnaphthalene in the aliphatic fraction exceeds 5% of the total concentration for naphthalene or 2-methylnaphthalene in the LCS or LCSD, fractionation must be repeated on all archived batch extracts.**

**NOTE:** The total concentration of naphthalene or 2-methylnaphthalene in the LCS/LCSD pair includes the summation of the concentration detected in the aliphatic fraction and the concentration detected in the aromatic fraction.

Comments: Concentration in the aliphatic fraction < 5% of the total  
concentration for naphthalene and 2-methylnaphthalene

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4. **Fractionation Check Standard** – A fractionation check solution is prepared containing 14 alkanes and 17 PAHs at a nominal concentration of 200 ng/μl of each constituent. The Fractionation Check Solution must be used to evaluate the fractionation efficiency of each new lot of silica gel/cartridges, and establish the optimum hexane volume required to efficiently elute aliphatic hydrocarbons while not allowing significant aromatic hydrocarbon breakthrough. For each analyte contained in the fractionation check solution, excluding n-nonane, the Percent Recovery must be between 40 and 140%. A 30% Recovery is acceptable for n-nonane.

Is a fractionation check standard analyzed?

Yes? or No?

Comments: Not applicable.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### XII. QUANTITATION LIMITS AND SAMPLE RESULTS

The sample quantitation evaluation is to verify laboratory quantitation results.

In order to demonstrate the absence of aliphatic mass discrimination, the response ratio of C28 to C20 must be at least 0.85. If <0.85, this nonconformance must be noted in the laboratory case narrative.

The chromatograms of Continuing Calibration Standards for aromatics must be reviewed to ensure that there are no obvious signs of mass discrimination.

Is aliphatic mass discrimination observed in the sample? Yes? or No?

Is aromatic mass discrimination observed in the sample? Yes? or No?

1. In the space below, please show a minimum of one sample calculation:

FA40754-2 EPH (C19 – C36, Aliphatics)

RF =  $1.074 \times 10^6$

FID

[ ] =  $182835695 / 1.074 \times 10^6$

= 170.2 ppm Ok

## DATA REVIEW WORKSHEETS

2. If requested, verify that the results were above the laboratory method detection limit (MDLs).
3. If dilutions performed, were the SQLs elevated accordingly by the laboratory? List the affected samples and dilution factor in the table below.

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION

If dilution was not performed, estimate results (J) for the affected compounds. List the affected samples/compounds:

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